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EMBRITTLEMENT OF GUN STEEL BY LIQUID LEAD

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A study has been made of the fracture behavior of gun steel in liquid lead and inert argon environments at 630°K in monotonic, static and cyclic fatigue test conditions. Smooth and notched specimens tested under the above conditions were severely embrittled by liquid lead and failed by intergranular mode as compared to a ductile mode in argon. Smooth specimens tested monotonically and in static fatigue failed in a catastrophic manner near the yield

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stress of the steel. Specimens with machined notches and with a fatigue crack at the root of the notch tested in liquid lead failed at the stress intensity values of 35 Ksi $\sqrt{in}$ and 7 Ksi $\sqrt{in}$ static fatigue and cyclic fatigue tests respectively, whereas in inert argon environment fatigue precracked specimens failed in cyclic fatigue at 135 Ksi $\sqrt{in}$. The susceptibility to embrittlement of steel specimens in liquid lead tested in cyclic fatigue was the same whether the notch was as machined or had a fatigue precrack at the root of the notch, i.e., embrittlement was independent of the sharpness of the root radii. These and other results are discussed in terms of the prevalent "reduction in cohesion" mechanism of liquid metal embrittlement proposed by Westwood and Kamdar. The critical conditions and prerequisites for the occurrence of embrittlement of gun steel in liquid lead are also discussed.

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INTRODUCTION

Lead electroplated inside the bore of a gun tube has historically been used as a lubricant for the mechanical swage autofrettage process. The lead plated tubes are given a post-autofrettage thermal treatment at 630°K for four hours. Subsequent inspection of the gun tubes revealed occasional transverse cracks. In instances, these cracks were around the entire circumference and nearly through the wall thickness and resulted in complete failure during subsequent gun straightening (Figure 1). The fracture surface was brittle intergranular and was coated with a thin layer of lead. The swage autofrettage process produces high longitudinal residual tensile stresses in the bore surface of the gun and additionally produces good intimate contact between the lead and the steel. The presence of lead in intimate contact combined with tensile residual stresses and post-thermal treatment exceeding the melting temperature of lead raises the possibility that liquid lead embrittlement of steel may occur.

Liquid lead is known to embrittle steel tested in tension.[1] However, little is known about the effects of steady state (static fatigue) and cyclic fatigue loading on the susceptibility of steel to embrittlement. In the present investigation, the effects of liquid lead on the initiation and the propagation of cracks in gun steel under monotonic, static and cyclic tensile loading conditions were examined

[1]Gordon, P., Breyer, N., Broutman, L., Albright, D., Warke, W., (1969-73), "Environment Sensitivity of Structural Materials: Liquid Metal Embrittlement," Project Themes Progress Reports, Illinois Institute of Technology, Chicago, IL.

The results are discussed with regard to post thermal treatment transverse cracking of lead plated gun tubes and also in terms of the "reduction in cohesion" model of liquid metal embrittlement proposed by Westwood and Kamdar.

EXPERIMENTAL

Material used in this investigation was high strength 4335 gun steel (Table 1) and high purity (99.9%) lead. Smooth round tensile specimens 0.25" in diameter and with 1.25" gage length were machined from the steel and were used for crack initiation studies in monotonic and static fatigue tests. Single edge notched fracture toughness specimens having the following ASTM configuration were used for crack propagation studies in static and cyclic fatigue tests. The test specimens were 0.25" thick, 1.25" wide and had loading pinholes 4" apart. They had a 60° notch with 0.005" root radius extending 0.525" through the width of the specimen. Both the smooth and the single edge-notched specimens were cleaned in acetone, reverse etched and using the procedure described elsewhere[2] they were electroplated with a thick lead coating on the entire gage length including the notch of the single edge-notched specimens. Some of the specimens with and without lead were cycled at 300°K and a fatigue crack ~ 0.03" to .104" long was introduced at the root of the notch. During fatigue pre-cracking visible cracks were not observed in the lead plating indicating that the fatigue precrack was not exposed to air. Presumably the crack

[2] Sadak, J., Lead Plating Procedure, Private Communication, Watervliet Arsenal, Watervliet, NY.

surfaces and the tip are clean, unoxidized and therefore will be wetted by molten lead upon heating. A stainless steel envelope was spot welded to these specimens below the lead plated gage section and was filled with molten lead. This assured an adequate supply of lead for the test and prevented liquid lead from dripping off the surface of the specimen during cyclic fatigue tests. Smooth specimens and the notched specimens without fatigue precrack were mounted in a Rhiele tensile machine and were enclosed in a furnace. The single edge notch bars with notches or with a fatigue precrack and with or without lead were mounted in a Sontag fatigue testing machine and enclosed in a split furnace. The monotonic and static fatigue tests were carried out in the Rhiele tensile testing machine whereas the cyclic fatigue tests were performed in the Sontag fatigue machine respectively.

PROCEDURE

(a) Monotonic and Static Fatigue

First the smooth and notched (as machined) lead plated specimens were heated to 630°K in flowing argon atmosphere. Then they were tested in tension monotonically to failure and the load elongation curves recorded. Subsequently, the specimens were cooled in argon to room temperature. In static fatigue tests, smooth specimens and notched specimens with or without fatigue precrack and with or without lead coatings were preloaded at 300°K to various stresses and heated to 630°K in argon atmosphere while maintaining the stress constant. At 630°K, the stress on the specimen was kept constant for four hours

and the time to failure or otherwise was noted.

(b) Cyclic Fatigue Test

The single edge notched specimens with and without a fatigue precrack and with or without lead coatings were used in cyclic fatigue tests. To facilitate wetting of the fatigue precrack and the root of the notch by lead, the specimens were fatigued in tension at 600 lb tensile load for one thousand cycles in liquid lead at 630°K. This procedure did not nucleate a crack at the root of the notch or result in crack growth or fatigue failure of the specimen. The specimens were then loaded in tension to various loads and tested to failure in a Sontag fatigue tester at 1800 rpm in liquid lead or argon environments. The cycles to failure were noted and the specimen was cooled in argon to 300°K. The lead from the fractured surfaces of the broken specimen was removed by heating in vacuum and a subsequent chemical treatment. The length of crack at failure was measured and the fractured surfaces were examined in a scanning electron microscope for failure mode, etc.

(c) Other Tests

The monotonic, static and cyclic fatigue tests were also carried out in the liquid lead environment containing 5 w/o antimony to investigate the effects of antimony additions - antimony being the major impurity found in lead - on the susceptibility of lead to embrittlement.

RESULTS

The tensile and static fatigue test data for crack initiation in a smooth specimen in argon and liquid lead environments are given in Tables 1 and 2(a). The specimens tested in argon failed in a ductile manner whereas those tested in liquid lead failed by brittle intergranular mode with total loss in ductility, (Tables 1 and 2(a) and Figures 2 and 3).

TABLE 1. TENSILE TEST DATA FOR 4335 STEEL TESTED IN TENSION IN LIQUID LEAD AND ARGON ENVIRONMENTS AT 630°K

Specimen	Environment	Yield Stress (Ksi)	Fracture Stress (Ksi)	R.A. (%)	Fracture Mode
Smooth Tensile	Argon	125	515	70	Ductile
Smooth Tensile	Liq. Lead	125	120	0	Brittle

TABLE 2(a) and (b). STATIC FATIGUE TEST DATA FOR 2(a) SMOOTH AND 2(b) AS NOTCHED AND FATIGUE PRECRACK AT ROOT OF NOTCH SPECIMENS LOADED IN LIQUID LEAD AND ARGON ENVIRONMENTS AT 630°K

2(a)

Specimen	Environment	Stress (Ksi)	Time to Failure at 630°K	Fracture Mode
Smooth	Argon	120-150	No failure (4 hrs)	-
Smooth	Liq. Lead	100	10 min.	Brittle
Smooth	Liq. Lead	90 Ksi	No failure (4 hrs)	-

2(b)

Specimen	Environment	Stress Intensity Ksi$\sqrt{in}$	Time To Failure at 630°K	Fracture Mode
Fatigue Precracked	Argon	135	3-4 hours	Ductile
Notched - no fatigue crack	Argon	135	No failure	-
Notched - no fatigue crack	Liq. Lead	35		Brittle

Static fatigue results for notched specimens given in Table 2(b) show that the smallest stress intensity or K value for failure in liquid lead is 35 Ksi$\sqrt{in}$. This value is four times lower than the stress intensity for fracture of 135 Ksi$\sqrt{in}$ for steel in an inert argon environment.

The cyclic fatigue results for specimens with notches and with a fatigue crack at the root of the notches tested in liquid lead and argon at 630°K are given in Tables 3(a) and (b).

TABLE 3(a). CYCLIC FATIGUE TEST DATA FOR SINGLE EDGE NOTCHED SPECIMENS FATIGUE PRECRACKED AT 300°K AND TESTED AT 1800 RPM AND 630°K IN LIQUID LEAD AND ARGON ENVIRONMENTS

Tensile Load (Lbs)	Environment	Cycles To Failure	Stress Intensity Ksi$\sqrt{in}$	Fracture Mode
1950	Argon	2×10^5	135	Ductile
1950	Liq. Lead	2×10^3	15	Brittle
800	Liq. Lead	2×10^3	7	Brittle

TABLE 3(b). TEST CONDITIONS SAME AS IN TABLE 3(a) EXCEPT THAT THE SPECIMENS WERE IN AS NOTCHED CONDITION WITH NO FATIGUE CRACK AT THE ROOT OF THE NOTCH

Tensile Load	Environment	Cycles To Failure	Stress Intensity Ksi$\sqrt{in}$	Fracture Mode
1950	Argon	5×10^5 (no failure)	-	-
1950	Lead	2×10^3	15	Brittle
1250	Lead	2×10^3	11	Brittle
800	Lead	2×10^3	7	Brittle
500	Lead	2×10^5 (no failure)	5	Threshold Value

The initial stress intensity at the root of the notch or the tip of the fatigue precrack at the point of failure reported above and for the cyclic tests was calculated using measured crack length a, the width of the specimen b, the applied tensile stress σ, the equation of $K = \sigma\sqrt{\pi a}\, F(a/b)$ and the calibration curve for values of F for given values of a and b. The calibration curve and the equation for calculating K values for single edge notched precracked flat bar specimens tested in tension were taken from ASTM publication #410.[3,4] The results in Tables 3(a) and (b) show that the K values in liquid lead decreased by one order of magnitude to 7 Ksi$\sqrt{in}$ and cycles to failure decreased by more than two orders of magnitude (to two thousand cycles) as compared to those in argon.

[3]Brown, W. F. and Strawley, J. W., (1960) "Plain Strain Crack Toughness Testing of High Strength Metallic Materials," ASTM Technical Publication No. 410.

[4]Kamdar, M. H., July 1976, Watervliet Arsenal Technical Report WVT-TR-76029, "Environmental Effects of a Stearate Coating on the Fracture Behavior of Gun Steel."

The K values in liquid lead are the same for specimens with machined notches only or with a sharp fatigue precrack at the root of the notch. This is a significant result. The threshold value for embrittlement was 5 $Ksi\sqrt{in}$.

OTHER RESULTS

The test results for liquid lead containing 5 w/o antimony were the same as those reported for pure liquid lead. Thus, antimony had no additional effect on the embrittlement behavior of 4335 steel.

DISCUSSION

The smallest stress to initiate a crack in smooth specimens wetted with lead and tested in a tension or in static fatigue is the same as the tensile yield stress of the steel, Tables 1 and 2(a). This suggests that the simultaneous presence of both plastic flow due to yielding, a tensile stress in the specimen, and liquid lead in intimate contact with steel are necessary for brittle crack initiation. These results or conditions are the same as the general prerequisites for the occurrence of liquid metal embrittlement proposed by Kamdar[5]. Additionally, the result that yielding must precede fracture suggests the following mechanism for crack initiation in a liquid lead environment. Slip bands produced upon yielding are blocked at grain boundaries inducing high stress concentrations at the grain boundary barriers. Adsorption of the liquid lead atoms at the grain boundary reduces the cohesive strength of the iron-iron bonds in the boundary and as a consequence a lower stress concentration is required for

[5]Kamdar, M. H., (1973), "Embrittlement by Liquid Metals," Prog. Mat. Sci., 15, 289.

crack initiation. A brittle crack initiates in liquid lead environment when the stress concentration at the grain boundary induced by plastic deformation is equal to or greater than the reduced cohesive strength of steel.

In cyclic fatigue tests the stress intensity at fracture is one order of magnitude less than that in argon, Tables 3(a) and (b). Furthermore, this value (7 Ksi$\sqrt{in}$) is half an order of magnitude lower than that determined in static fatigue tests. The lower susceptibility to embrittlement in a static fatigue test may be caused by wetting of the crack tip only where the thin oxide film which is invariably present is broken by plastic deformation produced at the tip by the applied static load. Embrittlement occurs only where the crack tip is wetted by liquid lead. Thus, partial wetting of the crack tip will increase the fracture stress and decrease the susceptibility to embrittlement. On the other hand, intense slip steps are produced continuously at the crack tip during cyclic fatigue tests. The emergence of slip steps will break the oxide film allowing liquid lead to wet the newly created clean surfaces of the slip steps and the entire crack tip. Complete wetting indicates that all atoms at the crack tip will interact with the liquid metal atoms causing maximum susceptibility to embrittlement. Thus, the lowest value of K, 7 Ksi$\sqrt{in}$ in cyclic fatigue may constitute the threshold stress intensity for embrittlement of steel by liquid lead.

It is significant to note that for a sharp fatigue crack or a blunt machined notch the stress intensity at fracture and the number of cycles to failure are the same i.e., embrittlement is independent of the sharpness of the crack front or tip radii. As one would expect, the contrary is true for specimens tested in inert argon atmosphere, Tables 3(a) and (b). This behavior may be explained by following Westwood and Kamdar[6] who have suggested that embrittlement is a highly localized event occurring on an atomic scale only at the crack front. Thus for a brittle crack to initiate in liquid metal environment, the only important consideration should be that the strength of the metal-metal atom bonds along the very crack front be reduced by the adsorbed liquid metal atoms. Crack propagation does not depend upon the bluntness of the machined notch or similar crack blunting conditions produced for a sharp crack by plastic deformation occurring in the vicinity or away from the tip due to applied stress and is additionally independent of the liquid metal atoms adsorbed elsewhere near the crack front or on the crack surfaces. Thus, the embrittling action is highly localized and is independent of the radius of the crack tip. Occurrence of embrittlement or otherwise will depend upon the stress concentrations at the crack front only. For stress concentrations equal to or greater than the reduced cohesive strength of the metal atom bonds by the liquid metal atoms, the crack will propagate in a brittle manner whereas for lower stress concentrations a ductile behavior may be observed. It is not possible to calculate or even estimate the theoretical cohesive strength of

[6]Westwood, A. R. C., and Kamdar, M. H., (1963), Phil. Mag., 6, 787.

metals in the presence of adsorbed liquid metals[5]. Hence it is suggested that for the crack tip radii used in this investigation, the stress concentrations at the tip may be considered equal to or greater than the reduced cohesive strength of the iron-iron bonds by the adsorbed liquid lead atoms making embrittlement susceptibility independent of the crack tip radii. It is possible of course that when a crack front is severely blunted, a brittle to ductile transition may occur in liquid metal environments. Such transitions for embrittlement couples have been observed and are reported in a recent review by Kamdar.[5]

SUMMARY

(1) 4335 Steel is severely embrittled by liquid lead when tested in tension in monotonic, static and cyclic fatigue tests.

(2) Yielding is necessary for crack initiation in steel in liquid lead environment.

(3) Severe embrittlement of 4335 gun steel in static fatigue and cyclic fatigue tests suggests that a definite possibility exists for the initiation of a brittle crack at very low tensile residual stresses from inclusions or defects wetted with lead during thermal stress relief heat treatment of lead coated autofrettage gun tubes at 630°K.

(4) The lead embrittlement of steel is independent of the crack tip radii of order 0.005" or less.

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[5] Kamdar, M. H., (1973), "Embrittlement by Liquid Metals," Prog. Mat. Sci., 15, 289.

(5) The "reduction in cohesion" model of liquid metal embrittlement is used to explain the results (2) and (4) above.

(6) Additions of antimony to lead does not increase or decrease the embrittlement susceptibility of steel.

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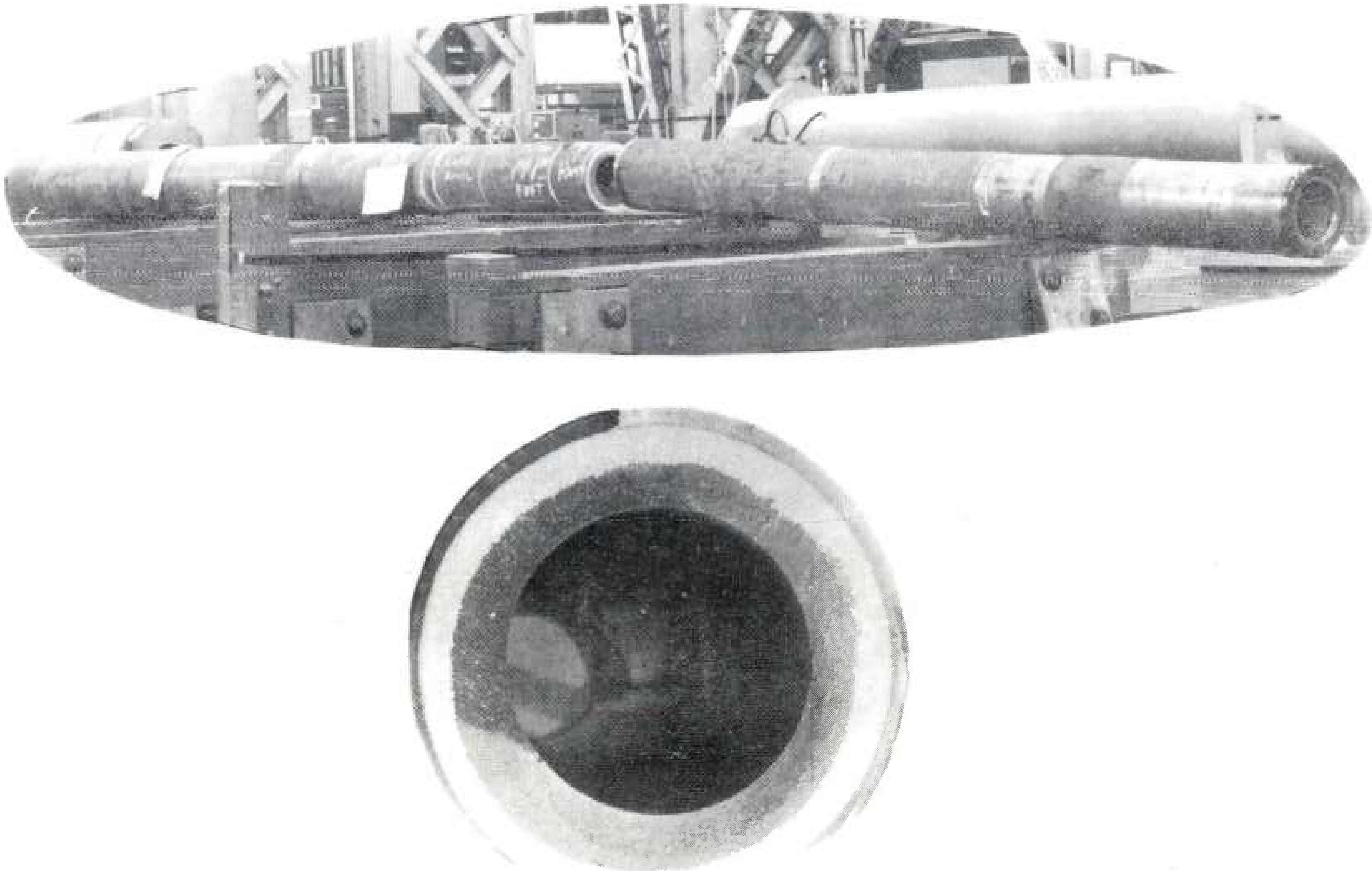

Figure 1. Lead coated 105mm gun tube which broke brittlely by transverse cracking in two pieces after heat treatment at 675°F for 8 hours. The transverse cracked cross section is shown below.

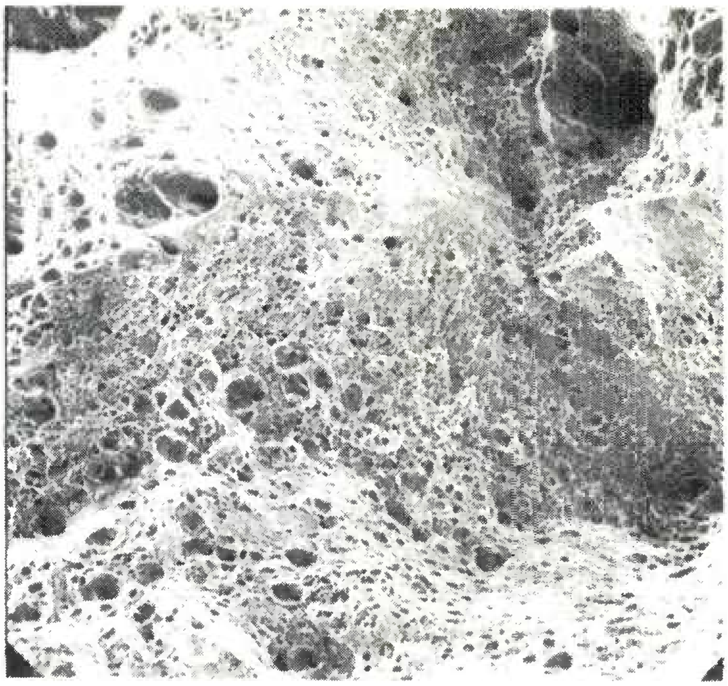

Figure 2. Smooth specimen tested in argon showing ductile failure. 500X

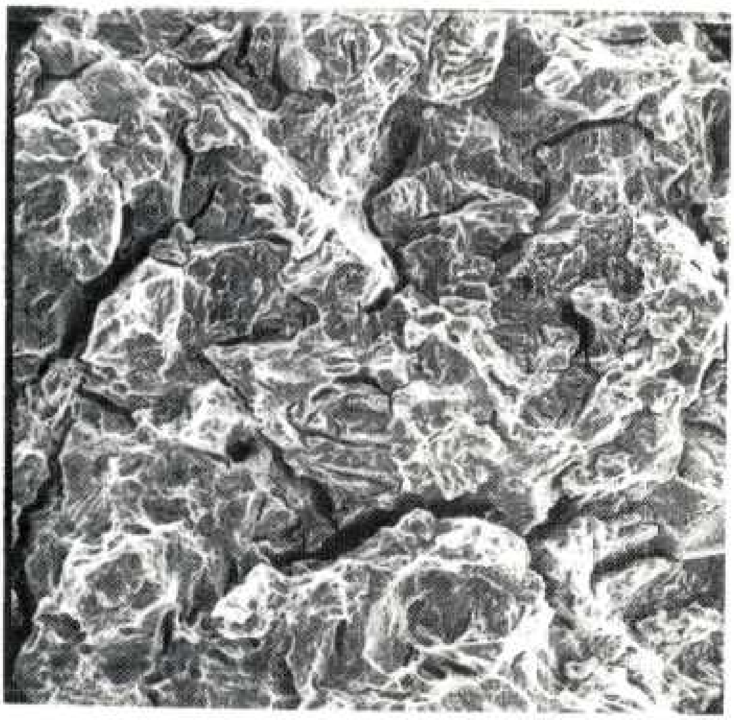

Figure 3. Smooth specimen tested in liquid lead showing brittle intergranular failure. 500X

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